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Passive Badge Assessment for Long-term, Low-level Air Monitoring on Submarines: Chamber Validation

Kimberly P. Williams

Nova Research, Inc.
Alexandria, VA

Susan L. Rose-Pehrsson

Chemical Dynamics and Diagnostics Branch
Chemistry Division

David A. Kidwell

Surface Chemistry Branch
Chemistry Division

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14. ABSTRACT
Exposure chambers were designed and engineered for validating passive diffusion badges for long-term atmosphere monitoring onboard U.S. Navy nuclear submarines. This is a validation study of the reproducibility among five identical chambers. Long-term sampling was evaluated for a 21-day period by comparing the response of a passive badge to an active tube sampling method, while being simultaneously exposed to homogeneous test vapors. In this study, the badges continued to accumulate the analyte for the entire 21-day sampling period. The average relative standard deviation among badges was 7% and 5.7% for tubes, demonstrating reproducibility among the chambers. The chambers are recommended for future use for analyte-specific, passive badge validation testing.

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Submarine atmospheric monitoring; SAHAP; Passive sampling; Passive badges; Formaldehyde; Air samples; NIOSH methods; U.S. Navy OEL; Contamination levels

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Contents

PASSIVE BADGE ASSESSMENT FOR LONG-TERM, LOW-LEVEL AIR MONITORING ON SUBMARINES: CHAMBER VALIDATION

1.0 Introduction

The submarine is a unique working and living environment, as submariners are contained in this environment 24 hours a day for the duration of deployment. It is important to know and monitor the safety of the atmosphere to which they are exposed. Current methods of air monitoring onboard U.S. Navy (USN) nuclear submarines include the central atmosphere monitoring system (CAMS) and active tube sampling (Draeger). The CAMS provides continuous, real-time air analysis for only a few critical compounds. Draeger tubes provide real-time results for other species of interest, but sampling is not continuous. The Draeger tube methods are labor intensive and have poor reproducibility as the result of a manually operated hand pump, as well as multiple interpretations of the manually read tube results. Implementing passive badges would greatly reduce sources of error, as they are professionally analyzed and require very little human manipulation. They may supplement or even replace certain sampling procedures while providing continuous air sampling, relieving the sailors to perform other important duties onboard the ship. Additionally, numerous analytes can be tested at the same time using one or multiple badges.

For use on submarines, passive badges should provide continuous air monitoring for up to 28 consecutive days. Before the badges can be used in this application, they must be validated for long-term use, as they are currently only validated commercially for a normal 8-hour working day. To assess their long-term responses, the badges were compared to commonly-used active sampling tubes for up to a 28-day exposure. An exposure chamber was designed to provide a homogeneous test vapor to both the tubes and the badges. Six of these chambers were manufactured. Validation of the chambers was necessary to be able to use the chambers interchangeably and be confident of obtaining reproducible results. Formaldehyde was used as the test vapor to validate the chambers.

2.0 Experimental

Formaldehyde badges were supplied by Assay Technology, Inc. (# 571-Aldehydes). The badges used dinitrophenylhydrazine (DNPH), coated onto a fiberglass pad, as a capture and derivatizing agent for formaldehyde. The same chemistry was used on the active sampling tubes, SKC LpDNPH S10. The formaldehyde-DNPH derivative was extracted then measured by HPLC (HP 1100) and quantified against a 6-point calibration curve, prepared by making volumetric dilutions of a stock standard, Restek #31808 Aldehyde/Ketone DNPH Standard. Certified gas cylinders (Airgas 15 ppm and 35 ppm) provided the formaldehyde vapor, which was diluted into a clean, humidified, airstream to provide the desired test vapor concentration, 4 ppb (10% USN 90-day limit). The gas

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cylinder concentrations were independently verified before being used. Active tube samples were collected using a sample pump, SKC pocket pump 210-1002, to pull approximately 50 mL/min of test vapor through each tube.

2.1 Design of the Chambers

The test chambers were designed for the purpose of delivering a reproducible, homogenous test vapor, while simultaneously accommodating five passive badges and five active tubes. The chambers are comprised of multiple parts: introduction chamber, mixing baffles, badge plate, tube ports, and a fan, as shown in Figure 1. The chamber's body is tubular, chosen over a traditional rectangular shape to reduce "dead" air space within corners of the chamber. The body is 10.8 cm in diameter (ID) and 30.5 cm long. A plate within the chamber was designed to hold five badges, each being exposed to a uniform airstream at a specified face velocity, as shown in Figure 2. The sampling rate of the aldehyde badge, as specified by the manufacturer, was 13.1 mL/min. To maintain this sampling rate, a minimal linear face velocity of $\geq$17 cm/sec, or 13 L/min, was sustained (1). The plate directed a total volume of 30 L/min of test vapor through the 1 cm × 2.5 cm openings in front of each of the five badges, providing the appropriate face velocity. The fan at the back of the chamber pulled the test vapor through the chamber as it was introduced, at approximately 29 L/min. A slight overpressure in the chamber prevented room air from leaking into the system. Two baffles at the front of the chamber aided in mixing the vapor stream.

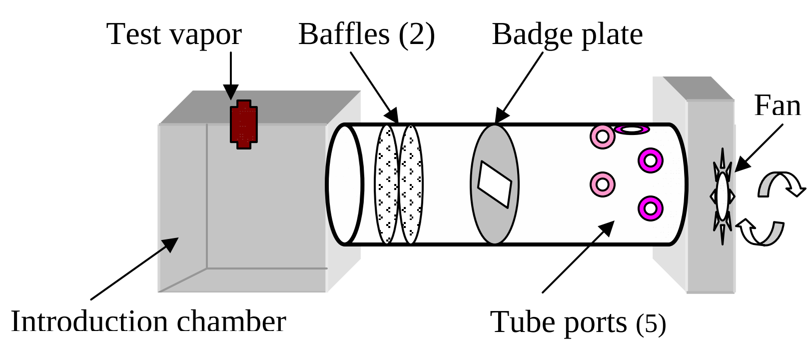

Figure 1. Illustration of a validation chamber.

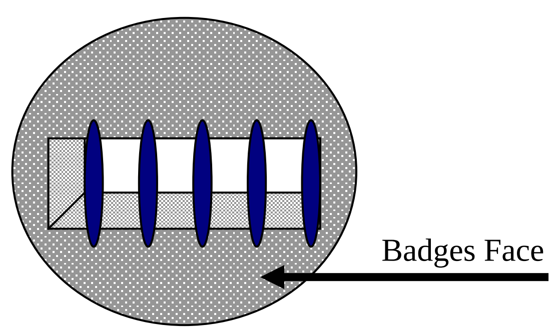

Figure 2. Badge plate filled with 5 badges.

2.2 Experimental Design

Using six chambers required an excess of 180 L/min of clean, humidified air. The 180 L/min was obtained by passing compressed, house-air through two molecular sieves, two humidifying tanks, then through four mass flow controllers and into a vapor distribution manifold, as shown in Figure 3. The molecular sieves removed moisture and carbon dioxide (CO_2) from the air. This "clean" air was humidified by passing the air through a pressurized tank filled with deionized water.

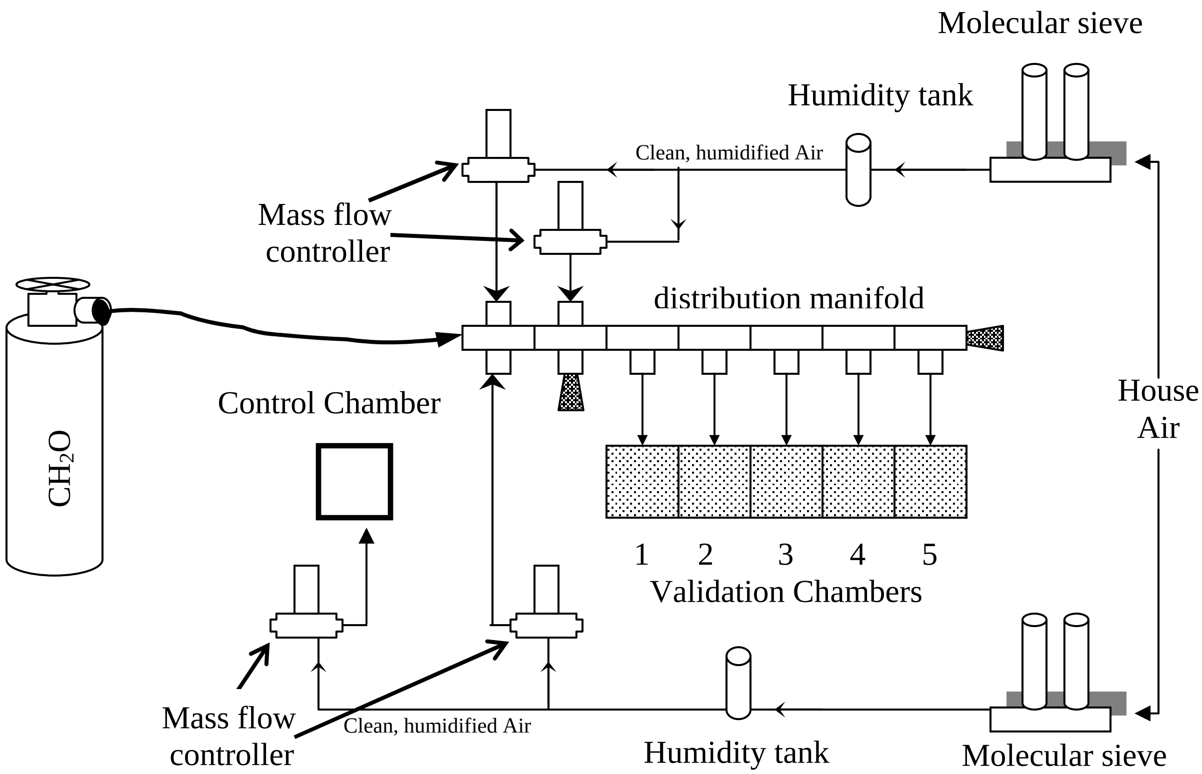

Figure 3. Validation system setup.

The distribution manifold was made of several PVC units that were purchased from a local hardware store and constructed using methylene chloride glue. The formaldehyde (CH_2O) gas stream was introduced at the front of the manifold, where mixing occurred before being distributed among the chambers. Several samples were taken at multiple locations along the manifold, to assure that the PVC did not affect the test vapor concentration and that proper mixing of the vapor had been achieved. No significant change of formaldehyde concentration was observed. There were also no additional matrix interferences, or "garbage," seen in the chromatograms indicative of the test vapor or apparatus. Five Teflon tubes, of equal length and diameter, evenly distributed 30 L/min of the formaldehyde test vapor to each of five of the exposure chambers. The sixth chamber analyzed 30 L/min of clean air only, provided by a separate flow controller, and was used as the control. The badges were inserted into the badge plate, all badges facing

the same direction. Active sampling of the tubes was provided by a single pump per chamber, set to pull a total of 250 mL/min. The total volume drawn was distributed among the five sampling tubes, via equal length Tygon tubing from another PVC mini-manifold, to provide a nominal sampling rate of 50 mL/min. The analyte interacted with the badge and tube sample substrates before contacting any Tygon tubing in the system. Due to slight differences in the tubes, as a result of manufacturing processes, the pressure drop across the tubes varied resulting in small variations of flow through the tube. The flow rate of each tube was measured independently using a Sierra mass flow meter before being inserted into the chamber and again each week until its removal. The average flow rate, per tube, was used when analyzing the final data results.

The chambers were filled with their respective tubes and badges. The pocket sampling pumps were turned on and individual flow rates of the tubes were measured. The flow rate of the formaldehyde gas was measured and used to verify that the proper vapor concentration was being administered to the chambers. The exposure began when the formaldehyde gas was introduced to the distribution manifold. The analyte exposure was nearly continuous for 28 days. The formaldehyde gas concentration was verified again following the weekly exchange of tubes and badges. The airflow was also recorded.

To monitor the progress of the experiment, a scheduled number of badges and tubes were systematically removed per week, as shown in Figure 4. Each week half of the tubes were removed from each chamber and analyzed, and new tubes were inserted in their place. Since there are five tubes, three tubes were removed the first week, then two tubes the next week. This pattern was repeated for the duration of the validation. Simultaneously, one badge was removed from each chamber and analyzed per week. A new badge was inserted in its place. At the end of Week 4, two of the original badges were removed from each chamber, instead of one. The gas analyte was turned off just prior to the sample exchange and turned back on following the exchange. The data was catalogued each week and used to compile a final data analysis at the end of the 28-day testing period. At the end of the 28 days, all of the remaining tubes and badges were removed from the chambers and analyzed, providing a compilation of data consisting of 13 tubes and 8 badges per chamber.

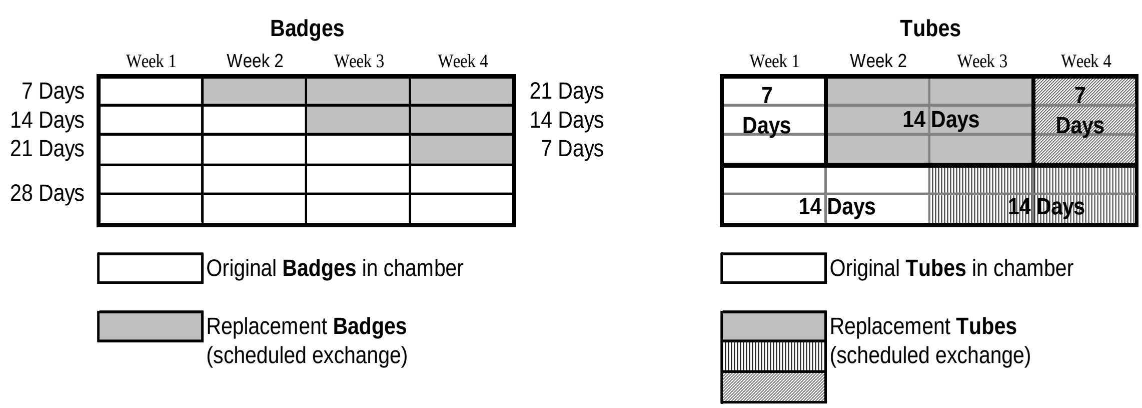

Figure 4. Schedule matrix of badge and tube exchanges, per chamber.

2.3 Analysis

The tube and badge samples were solvent-extracted and analyzed by HPLC following the NIOSH 2016 analytical method (2). Chromatograph specifications included a Restek Ultra C18, 5 µm, 150 x 4.6 mm reversed phase column, 40:60 water/acetonitrile mobile phase (isocratic), and a 20 µL sample loop injection. The method run time was 5 minutes with an elution rate of 1.5 mL/min. The retention time of the formaldehyde-DNPH derivative was approximately 2.6 minutes and the excess DNPH at 1.6 minutes. The formaldehyde-DNPH absorbance was read at 365 nm. An external calibration curve, made up from certified DNPH standards, was used for calculating concentrations.

2.3.1 Active Sampling Tubes

The tube cartridges were extracted using a SPE vacuum manifold. The tubes were set up on the manifold, with the flow valves closed. The vacuum was turned on and the flow valves opened to allow 10 mL of acetonitrile solvent to elute through each tube at a rate of approximately 5 mL/min. The eluent was collected into a clean sample vial then analyzed by HPLC.

2.3.2 Passive Badges

The back of the badge was removed to acquire the fiberglass sample pad. The pad was transferred to a sample vial containing 10 mL of acetonitrile. The vial was capped and sonicated for 5 minutes. Since sonication resulted in break-up of the badge, the sample was then centrifuged and the supernatant was analyzed by HPLC.

3.0 Results and Discussion

Data was gathered and compiled on a weekly basis by removing a scheduled number of tubes and badges from each chamber. Calculations were based on weekly measurements of the gas analyte, airstreams, and sampling rates. The expected sampling rate of the badges was constant, whereas, the sampling rate of the tubes varied slightly due to variability in the packing of the absorbent material. The flow rates of the tubes were measured upon introduction to the system and again every week after an exchange took place. The final measurement was taken prior to the tube's removal from the chamber. The average of all flow measurements, per tube, was used when calculating the formaldehyde concentration accumulated by each tube. Tables 1 and 2 show the raw data for tubes and badges. The observed concentration was obtained from the standard curve (µg/mL), relevant to the analyte peak area given by HPLC. The total amount of analyte recovered by each sampler, as well as the sampling rate of each sampler, was used to calculate the representative concentration of vapor in each chamber during the respective sampling period. With all data expressed in the same manner, direct comparisons could be made.

Table 1. Raw data for the active sampling tubes.

The theoretical concentration is 4 ppb.

Blank box	total µg sampled	sampling rate, L/min	Conc in chamber, ppb	average ppb	%RSD
tube 1	0.0960	0.0295	0.264	0.2	22.6
tube 2	0.1077	0.0424	0.205		
tube 3	0.0699	0.0335	0.168		
tube 4	0.2096	0.0493	0.172	0.1	30.5
tube 5	0.1019	0.0371	0.111		
tube 1b	0.1630	0.0557	0.118	0.2	23.4
tube 2b	0.1135	0.0240	0.191		
tube 3b	0.1310	0.0337	0.157		
tube 4b	0.1281	0.0440	0.118	0.1	28.3
tube 5b	0.1543	0.0353	0.176		
tube 1c	0.0495	0.0319	0.126	0.2	27.2
tube 2c	0.1106	0.0448	0.199		
tube 3c	0.0611	0.0379	0.130		

Box 3	total µg sampled	sampling rate, L/min	Conc in chamber, ppb	average ppb	%RSD
tube 1	0.1630	0.0399	0.331	0.3	4.0
tube 2	0.1397	0.0370	0.305		
tube 3	0.1281	0.0329	0.315		
tube 4	2.7563	0.0396	2.812	2.9	6.2
tube 5	3.0502	0.0401	3.071		
tube 1b	4.6918	0.0377	5.033	5.1	2.0
tube 2b	5.7134	0.0446	5.172		
tube 3b	5.2913	0.0408	5.236		
tube 4b	4.0980	0.0392	4.220	4.3	2.0
tube 5b	3.7720	0.0351	4.342		
tube 1c	1.8860	0.0366	4.164	4.1	0.7
tube 2c	2.3604	0.0465	4.106		
tube 3c	2.2819	0.0447	4.130		

Box 1	total µg sampled	sampling rate, L/min	Conc in chamber, ppb	average ppb	%RSD
tube 1	0.1543	0.0444	0.281	0.3	11.4
tube 2	0.1630	0.0374	0.353		
tube 3	0.1484	0.0364	0.330		
tube 4	3.5421	0.0506	2.826	2.9	2.0
tube 5	3.2569	0.0453	2.907		
tube 1b	6.2867	0.0506	5.023	5.1	1.6
tube 2b	6.3770	0.0497	5.184		
tube 3b	4.6336	0.0368	5.087		
tube 4b	4.0020	0.0334	4.841	4.8	2.6
tube 5b	4.5550	0.0394	4.667		
tube 1c	2.2440	0.0376	4.829	4.8	1.2
tube 2c	1.8278	0.0312	4.741		
tube 3c	1.9879	0.0341	4.718		

Box 4	total µg sampled	sampling rate, L/min	Conc in chamber, ppb	average ppb	%RSD
tube 1	0.1310	0.0337	0.315	0.3	1.9
tube 2	0.1805	0.0447	0.327		
tube 3	0.1484	0.0375	0.320		
tube 4	3.2045	0.0423	3.058	3.1	1.1
tube 5	2.7796	0.0362	3.105		
tube 1b	5.7163	0.0443	5.213	5.2	0.4
tube 2b	4.0194	0.0313	5.188		
tube 3b	5.6581	0.0442	5.176		
tube 4b	4.1300	0.0361	4.627	4.5	3.4
tube 5b	3.8594	0.0354	4.409		
tube 1c	2.3721	0.0448	4.279	4.3	1.8
tube 2c	2.7097	0.0494	4.432		
tube 3c	1.8016	0.0337	4.320		

Box 2	total µg sampled	sampling rate, L/min	Conc in chamber, ppb	average ppb	%RSD
tube 1	0.1630	0.0478	0.276	0.3	16.8
tube 2	0.2154	0.0452	0.385		
tube 3	0.1339	0.0335	0.323		
tube 4	2.6340	0.0368	2.892	2.8	3.7
tube 5	3.8565	0.0567	2.746		
tube 1b	4.6947	0.0356	5.323	5.4	2.4
tube 2b	5.0847	0.0370	5.547		
tube 3b	5.0469	0.0383	5.324		
tube 4b	3.3267	0.0307	4.373	4.4	1.8
tube 5b	5.2535	0.0473	4.487		
tube 1c	2.0956	0.0397	4.271	4.4	7.2
tube 2c	2.3401	0.0448	4.221		
tube 3c	1.8889	0.0318	4.800		

Box 5	total µg sampled	sampling rate, L/min	Conc in chamber, ppb	average ppb	%RSD
tube 1	0.1426	0.0413	0.279	0.3	3.3
tube 2	0.1601	0.0443	0.292		
tube 3	0.1397	0.0411	0.275		
tube 4	2.2033	0.0311	2.862	2.8	1.5
tube 5	2.0141	0.0290	2.803		
tube 1b	5.5242	0.0416	5.365	5.4	1.1
tube 2b	5.3554	0.0398	5.441		
tube 3b	6.7059	0.0494	5.488		
tube 4b	3.3937	0.0287	4.777	4.4	11.9
tube 5b	4.0107	0.0401	4.038		
tube 1c	2.0607	0.0395	4.221	4.3	1.3
tube 2c	2.0781	0.0390	4.306		
tube 3c	2.3546	0.0440	4.324		

The average flow rate of the air in the Blank box (control) was 28.81 L/min.
The average flow rate of the air in the test chambers was 34.93 L/min.
7 days = 10080 minutes

***** average concentration among the chambers for 28 days = 2.918 ppb
****** average concentration among the chambers, not considering samples in the chamber for WEEK 1 = 3.948 ppb
******* average %RSD, not considering WEEK 1 exposures = 2.8%

Table 2. Raw data for passive badges.

The theoretical concentration is 4 ppb. The badge sampling rate is 0.0131 L/min.

Blk Box	total µg sampled	Conc in chamber, ppb		
badge 1	0.1572	0.969		
badge 2	0.2299	0.709		
badge 3	0.2707	0.557		
badge 4	0.0873	0.135	average	%RSD
badge 5	0.0931	0.144	0.1	4.6
badge 1b	0.2503	0.515		
badge 2b	0.1048	0.323		
badge 3b	0.0582	0.359		

Box 1	total µg per sample	Conc in chamber, ppb		
badge 1	0.1572	0.969		
badge 2	0.7276	2.244		
badge 3	1.3825	2.843		
badge 4	1.3650	2.105	average	%RSD
badge 5	1.3214	2.038	2.1	2.3
badge 1b	1.5018	3.088		
badge 2b	0.9197	2.837		
badge 3b	0.3871	2.388		

Box 2	total µg per sample	Conc in chamber, ppb		
badge 1	0.1601	0.987		
badge 2	0.6549	2.020		
badge 3	1.0711	2.202		
badge 4	1.8103	2.792	average	%RSD
badge 5	1.3854	2.136	2.5	18.8
badge 1b	1.6299	3.351		
badge 2b	0.8935	2.756		
badge 3b	0.4017	2.478		

Box 3	total µg per sample	Conc in chamber, ppb		
badge 1	0.1484	0.916		
badge 2	0.6461	1.993		
badge 3	1.1497	2.364		
badge 4	1.4174	2.186	average	%RSD
badge 5	1.3971	2.154	2.2	1.0
badge 1b	1.3330	2.741		
badge 2b	0.8353	2.576		
badge 3b	0.3871	2.388		

Box 4	total µg per sample	Conc in chamber, ppb		
badge 1	0.1484	0.916		
badge 2	0.6374	1.966		
badge 3	1.1467	2.358		
badge 4	1.5309	2.361	average	%RSD
badge 5	1.3767	2.123	2.2	7.5
badge 1b	1.4320	2.944		
badge 2b	0.8149	2.513		
badge 3b	0.3696	2.280		

Box 5	total µg per sample	Conc in chamber, ppb		
badge 1	0.1397	0.862		
badge 2	0.6345	1.957		
badge 3	1.0332	2.124		
badge 4	1.3825	2.132	average	%RSD
badge 5	1.5280	2.356	2.2	7.1
badge 1b	1.4698	3.022		
badge 2b	0.8732	2.693		
badge 3b	0.4104	2.531		

The average flow rate of the air in the Blank box (control) was 28.81 L/min.
The average flow rate of the air in the test chambers was 34.93 L/min.
7 days = 10080 minutes

***** average concentration among the chambers = 2.2 ppb
****** average concentration among the chambers, not considering samples removed for WEEK 1 = 2.4 ppb

As a result of the sampling schedule matrix, badges and tubes were sampled for a different number of days. Tubes were sampled for 7 and 14-day durations, while badges were sampled for 7, 14, 21, and 28 days. Due to capacity limitations, active sampling tubes could not be collected for 28 days. To compare tubes to badges, the tube results were configured to reflect the same exposure times as the badges. For example, to compare 21-day badges, tube samples from week 1 (7 days) and from week 3 (14 days) were added together to obtain a 21-day result representative of the same exposure period as the badge. Collectively, the data were representative of the first 7, 14, 21, and 28 days and for the last 21, 14, and 7 days. For this experiment, however, the first 7 days were not included in the validation results, due to an error with the formaldehyde gas concentration. The first week of the experiment did not yield expected results. The concentrations in the compressed gas tanks were found to be different than the certified values. The tanks provided concentrations at 20% of their certified values. This was a

consistent trend among several tanks from various vendors. The discrepancy may have occurred as the result of the manufacturer using an improperly cleaned tank or possibly that formaldehyde is not stable as a gas and may have polymerized in the tank prior to use. As a result, the first week of sampling used a gas concentration too low to obtain adequate data. The problem was discovered at the beginning of Week 2 and the concentration of the gas stream was adjusted to provide the correct concentration. The gas stream was then verified each week for the remaining 21-day exposure period. As a result of the error that occurred during Week 1, the results were not considered in the final evaluation of this validation. The concentration values obtained during Week 1 were subtracted from all respective samples.

Data from the control "clean" chamber were good. All blanks were below 25% of the theoretical concentration, with an average of 4% recovery of formaldehyde on tubes and 12% on badges. This indicated that the molecular sieves were adequately cleaning the air and that foreign particles and material off-gassing were at a minimum. The fact that the spectra were not completely blank could be the result of minute contaminations, which likely occurred during manufacturing of the sampling media.

Accumulation of the analyte was consistently greater on the tubes than on the badges, see Figure 6. Badge results were typically 40% lower than tube results. The percent relative standard deviation (%RSD) of tubes among all chambers ranged from 3.0-9.8%, with an average of 5.7%. The %RSD of badges among all chambers ranged from 4.0-11.7%, with an average of 7.0%. The low RSD values indicate that the results among all chambers were consistent, see Table 3. Although tube results were typically higher than those of the badges, the relationship remained relatively constant. The difference may be explained by a difference in the badges' sampling rate. The sampling rate given by the manufacturer may be in error, as it is an experimentally determined figure for an eight-hour exposure period. Since the specifications of our test setup are different than that of the manufacturer, it is possible that the proposed sampling rate is merely a guideline without a finite definition and may change over time and with a changing concentration gradient. However, reproducibility among the chambers was demonstrated and is illustrated in Figure 7, showing a nearly linear relationship between the chambers. Figures 6 and 7 are only representative of the last 21 days of the validation. Since these samples were not affected by the error in Week 1, they are a more accurate representation of a 21-day response of the samplers.

Table 3. Reproducibility of tubes and badges when comparing the five chambers.

%RSD among tubes		%RSD among badges	
Week 1	9.81	Week 1	5.36
7 days	4.34	7 days	5.84
14 days	3.02	14 days	11.74
14 days	5.64	21 days	9.86
7 days	5.75	21 days	7.34
		14 days	4.90
		7 days	3.99

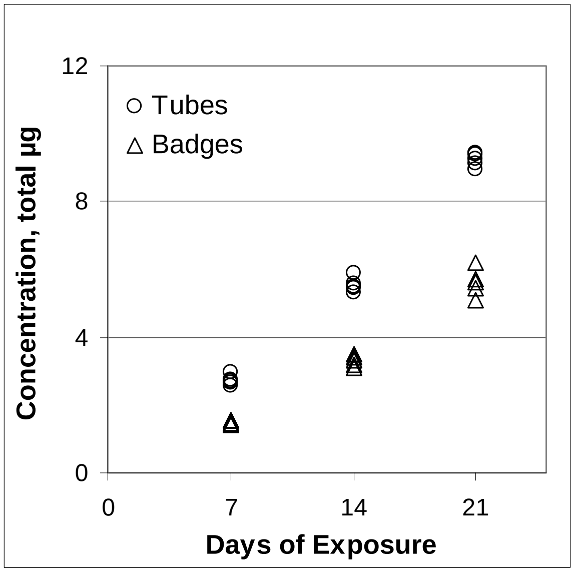

Figure 6. Accumulation of formaldehyde onto tubes and badges, at 4 ppb. Five data points are plotted per exposure period for tubes and badges. Each data point represents the data obtained from one of the five exposure chambers.

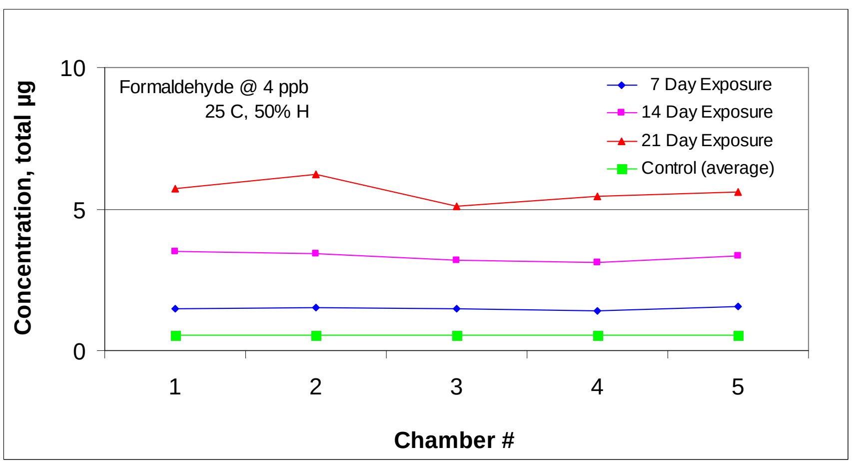

Figure 7. Reproducibility among the five exposure chambers.

4.0 Conclusions

The results provided by the five exposure chambers were compared to establish a reproducible correlation between the chambers. Reproducibility among active sampling tubes was demonstrated as well as among passive badges. Accumulation of the analyte onto tubes was consistently about 40% higher than accumulation onto badges. The average relative standard deviation of tubes was 5.7%. The average relative standard

deviation of badges was 7.0%. Average RSDs less then 10% indicate that the chambers can be used interchangeably and provide equivalent, reproducible results. The chambers are recommended for future use for analyte-specific badge validation testing.

5.0 References

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